O I P E JC67
JUN 1 8 2004
PATENT & TRADEMARK OFFICE

(19) JAPANESE PATENT OFFICE (JP)

(12) Publication of Unexamined Patent Application (KOKAI) (A)

(11) Japanese Patent Application Kokai Number: **H9-61142**

(43) Kokai Publication Date: March 7, 1997

(51) Int. Cl.[6]	Identification Symbol	JPO File No.	F1		Technical Indication
G 01 B 15/02			G 01 B 15/02	D	

Request for Examination: Not requested | Number of Claims: 7 | OL. | (5 pages total)

(21) Application Number: H7-221789

(22) Filing Date: August 30, 1995

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(54) [Title of the Invention] METHOD AND APPARATUS FOR MEASUREMENT OF FILM THICKNESS

(57) [Abstract]

[Object] [The object of the present invention is] to provide a method and apparatus for measuring and controlling, with monatomic layer thickness precision, the film thickness of a sample during film formation by vacuum deposition, by means of an apparatus construction which is inexpensive and which has a relatively simple structure.

[Constitution] [The apparatus] comprises a substrate 11, an electron gun 12 which is used to irradiate this substrate 11 with primary electrons, a vapor deposition apparatus 13 which deposits a sample on this substrate 11 by vapor deposition, and a secondary electron detector 14 which is used to measure the intensity of the secondary electrons that are knocked out by the primary electrons. The electron gun 12 has a function that allows the energy of the primary electrons to be freely set from approximately 10 eV to approximately 5 keV. In this case, the angle of incidence of the primary electrons on the sample is greater than the total-reflection angle.

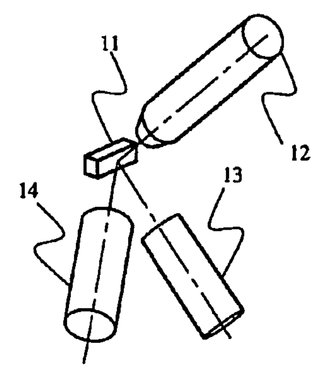


Figure 1

[Claims]

[Claim 1] A film thickness measurement method which is characterized by the fact that in a film thickness measurement method which utilizes the phenomenon whereby the intensity of the secondary electrons that are emitted as a result of the irradiation of the sample surface with primary electrons during the manufacture of a thin film sample by vacuum deposition oscillates with the period of the monatomic layer thickness as a function of the film thickness of the sample, the angle of incidence of the primary electrons as measured from the sample surface is set at an angle that is greater than the total-reflection angle of the electrons with respect to the sample.

[Claim 2] The film thickness measurement method according to Claim 1, wherein the oscillation of the absorption current into the sample that appears at the conditions under which the [above-mentioned] intensity oscillation of the secondary electrons is obtained is utilized.

[Claim 3] The film thickness measurement method according to Claim 1, wherein only secondary electrons having an energy at which the [above-mentioned] oscillation appears most clearly are used by selecting, by means of an energy analyzer, the energy of the secondary electrons for which the [above-mentioned] intensity oscillation measurement is performed.

[Claim 4] The film thickness measurement method according to Claim 2, wherein the energy of the primary electrons is set at an arbitrary value, and the energy of the primary electrons at which the secondary electron intensity oscillation or absorption current oscillation appears most clearly is selected and utilized.

[Claim 5] A film thickness measurement apparatus which is characterized by the fact that the apparatus comprises a substrate which is used for the formation of a thin film sample (on the surface of this substrate) by means of vacuum deposition, an electron gun which irradiates the surface of the above-mentioned sample with primary electrons, and a secondary electron detector which detects the secondary electrons that are emitted from the surface of the sample as a result of the above-mentioned irradiation with primary electrons, and the angle of irradiation of the surface of the sample with the primary electrons by means of the above-mentioned electron gun is set so that this angle is larger than the total-reflection angle as seen from the surface of the sample.

[Claim 6] The film thickness measurement apparatus according to Claim 5, wherein the oscillation of the absorption current into the sample is detected instead of the above-mentioned secondary electrons.

[Claim 7] The film thickness measurement apparatus according to Claim 5, which comprises an energy analyzer that is used to analyze the energy of the above-mentioned detected secondary electrons.

[Detailed Description of the Invention]

[0001]

[Field of Industrial Utilization] The present invention relates to a method and apparatus for measuring the film thickness of the sample during film formation by a vacuum deposition method such as molecular beam epitaxy with a precision corresponding to the thickness of a monatomic layer or better.

[0002]

[Prior Art] When a sample film is formed by a vacuum deposition method, it is conceivable that it might be possible to utilize the phenomenon whereby the intensity of the secondary electrons oscillates with the period of a monatomic layer thickness. This phenomenon has conventionally been observed under experimental conditions in reflective high-speed electron diffraction, and has been reported in *Journal of Crystal Growth*, Vol. 81 (1987), pp. 55-58.

[0003]

[Problems that the Invention is to Solve] In the above-mentioned method, however, it is necessary to cause the primary electrons to be incident at an angle that just grazes the sample surface (i.e., an angle that is approximately equal to or less than the total-reflection angle of the electrons as measured from the sample surface; below, all angles are assumed to be angles measured from the sample surface). Consequently, a special sample holder which does not interfere with the incidence of the electrons on the sample, and a mechanism for adjusting the angle of incidence of the primary electrons, are required in the sample holder or electron gun, so that the overall apparatus is increased in size and complexity. As a result, such a method suffers from the drawback of difficult handling of the apparatus and high cost of the apparatus as a whole.

[0004] The object of the present invention is to measure and control the film thickness of the sample during film formation by vacuum deposition with a precision corresponding to the thickness of a monatomic layer or better using an inexpensive apparatus which has a simple and compact structure.

[0005]

[Means for Solving the Problems] The above-mentioned problems are solved by setting the angle of incidence of the primary electrons on the sample at an angle that is greater than the total-reflection angle.

[0006]

[Operation] The intensity oscillation of the secondary electrons observed under experimental conditions in reflective high-speed electron diffraction in the prior art may be described as follows using the content of *Surface Science Letters*, Vol. 245 (1991), pp. L159-L162.

[0007] In cases where a sample in vacuum deposition is grown in layer form, the density of the step formed on the sample surface oscillates with the period of a monatomic layer. In cases where a step is present on the sample surface, the electrons can invade the interior of the sample via the end [of this step] even if the primary electrons are caused to be incident on the sample at an angle that is equal to or less than the total-reflection angle. Some of the invading electrons are directed toward the surface by diffraction in the interior portions of the sample; furthermore, some of these electrons are again directed toward the interior of the sample by total reflection at the sample surface, and are absorbed. Accordingly, if the step density oscillates, the number of electrons absorbed by the sample also oscillates. Since the number of secondary electrons is the difference between the number of primary electrons and the number of electrons absorbed by the sample, if the number of electrons absorbed by the sample varies, the secondary electron intensity also varies.

[0008] Accordingly, in conventional techniques, it has been believed that no oscillation of secondary electrons is observed in cases where the angle of incidence of the primary electrons on the sample is greater than the total-reflection angle.

[0009] However, according to the results of research conducted by the inventors, an oscillation of secondary electrons can be observed on the basis of the following principle even in cases where the angle of incidence of the primary electrons on the sample is greater than the total-reflection angle. Specifically, since the yield of secondary electrons increases with a decrease in the work function of the sample surface, the secondary electron intensity also oscillates if there is an oscillation in the work function. The oscillation of the work function of the sample surface during vapor deposition is explained in *Surface Science*, Vol. 298 (1993), pp. 173-186.

[0010] Furthermore, the same phenomenon may be explained as follows. Specifically, in cases where a sample is formed on a substrate by vapor deposition, quantum wells are formed in this sample if the sample is a thin film. This quantum well state is regulated by two sets of boundary conditions, i.e., the boundary conditions of the interface between the sample and the substrate

and the boundary conditions of the surface of the sample. Here, it would appear that the former boundary conditions do not depend on the film thickness of the sample; however, the surface shape (roughness) constituting the latter boundary conditions varies with the period of a monatomic layer in accordance with the step density, and the state density of the electrons within the thin film sample, and therefore the secondary electron intensity reflecting this state density, also varies with this period.

[0011] According to the results of research on this subject conducted by the inventors, the secondary electron oscillation during the vapor deposition of the sample on the substrate oscillates in synchronization with the period of the layer growth of the sample even in cases where the angle of incidence of the primary electrons on the sample is greater than the total-reflection angle; as a result, the film thickness of the sample during vapor deposition can be ascertained with a precision corresponding to the thickness of a monatomic layer or better. Here, there are no particular restrictions on the energy of the primary electrons, as long as this energy is greater than approximately 10 eV, which is the lowest energy at which the excitation of secondary electrons is possible.

[0012]

[Embodiments] Figure 1 is a diagram which shows a first basic construction of an embodiment of the film thickness measurement apparatus of the present invention. This construction comprises a substrate 11, an electron gun 12 which is used to irradiate this substrate 11 with primary electrons, a vapor deposition apparatus 13 which forms a sample on the surface of the substrate 11 by vapor deposition, and a secondary electron detector 14 which is used to measure the intensity of the secondary electrons that are knocked out [of the sample] by the primary electrons. Here, the electron gun 12 has a function that allows the energy of the primary electrons to be freely set at values ranging from approximately 10 eV to approximately 5 keV. Furthermore, there are no particular restrictions on the angle of incidence of the primary electrons on the sample, as long as this angle of incidence is greater than the total-reflection angle.

[0013] In this construction, irradiation with primary electrons having an energy of approximately 1 keV is performed by means of the electron gun 12 while a sample is formed on the surface of the substrate 11 by vapor deposition using the vapor deposition apparatus 13. In this case, the intensity of the secondary electrons that are emitted from the surface of the thin film sample formed on the substrate 11 is measured by the secondary electron detector 14. The measured intensity of the secondary electrons oscillates with the period of a monatomic layer as a function of the film thickness of the sample that is formed on the substrate 11. Figure 2 shows

the oscillation of the secondary electron intensity in a case where gold is deposited as the sample by vapor deposition. Since the period T of this oscillation is equal to the thickness of a monatomic layer of the sample, the thickness of the gold shown on the horizontal axis can be calibrated by T, so that the accurate thickness of the gold thin film can be ascertained. Accordingly, high-precision film thickness measurements can be made by using this phenomenon.

[0014] Furthermore, there may be cases in which the energy of the primary electrons at which the oscillation of the intensity of the secondary electrons appears most conspicuously varies according to the type and surface orientation, etc., of the sample. In the present embodiment, the energy of the primary electrons was set at approximately 1 keV as an example; however, the present method may be used under optimal conditions at which the oscillation of the intensity of the secondary electrons appears most conspicuously by varying the energy of the primary electrons in accordance with the type and surface orientation of the sample.

[0015] Figure 3 is a diagram which shows a second basic construction of an embodiment of the film thickness measurement apparatus of the present invention. This construction comprises a substrate 11, an electron gun 12 which is used to irradiate this substrate 11 with primary electrons, a vapor deposition apparatus 13 which deposits a sample on the surface of the substrate 11 by vapor deposition, an electron lens 31 which is used to focus the secondary electrons that are knocked out [of the sample] by the primary electrons and to transmit these secondary electrons in an efficient manner, an energy analyzer 32 which is used to discriminate the energy of the secondary electrons, and a secondary electron detector 14 which is used to measure the intensity of the secondary electrons following the energy analysis. Furthermore, as in the first embodiment, the electron gun 12 has a function that allows the energy of the primary electrons to be freely set at an energy ranging from approximately 10 eV to approximately 5 keV. Moreover, as in the first embodiment, there are no particular restrictions on the angle of incidence of the primary electrons on the sample, as long as this angle of incidence is greater than the total-reflection angle.

[0016] In this construction, irradiation with primary electrons having an energy of approximately 1 keV is performed by means of the electron gun 12 while a sample is deposited on the surface of the substrate 11 by vapor deposition by means of the vapor deposition apparatus 13. In this case, the secondary electrons that are emitted from the surface of the thin film sample formed on the substrate 11 are focused by the electron lens 31, and are efficiently transmitted to the energy analyzer 32. Only those electrons having a specified energy (among the secondary electrons that are incident on the energy analyzer 32) are conducted to the

secondary electron detector 14, and the intensity of these secondary electrons is measured. As in the first embodiment, the measured intensity of the secondary electrons oscillates as a function of the film thickness of the sample that is formed on the surface of the substrate 11, and since the period T of this oscillation is equal to the thickness of a monatomic layer of the sample, high-precision film thickness measurements can be made by monitoring this oscillation.

[0017] Furthermore, there may be cases in which the secondary electron energy range in which the oscillation of the secondary electron intensity appears most conspicuously varies according to the type and surface orientation, etc., of the sample. In the present embodiment, since it is possible to conduct only secondary electrons that have a specified energy to the secondary electron detector 14 by means of the energy analyzer 32, it is sufficient to select in this way the energy at which the oscillation of the secondary electron intensity appears most conspicuously, and to measure this secondary electron intensity.

[0018] Furthermore, as in the first embodiment, there may be cases in which the energy of the primary electrons at which the oscillation of the intensity of the secondary electrons appears most conspicuously varies according to the type and surface orientation, etc., of the sample. Accordingly, in this embodiment as well, the present method may be used under optimal conditions at which the oscillation of the intensity of the secondary electrons appears most conspicuously by adjusting the energy of the primary electrons in accordance with the type and surface orientation of the sample.

[0019] Figure 4 is a diagram which shows a third basic construction of an embodiment of the film thickness measurement apparatus of the present invention. This construction comprises a substrate 11, an electron gun 12 which is used to irradiate this substrate 11 with primary electrons, and a vapor deposition apparatus 13 which deposits a sample on the surface of the substrate 11 by vapor deposition. Furthermore, as in the first embodiment, the electron gun 12 has a function that allows the energy of the primary electrons to be freely set at an energy ranging from approximately 10 eV to approximately 5 keV. Moreover, as in the first embodiment, there are no particular restrictions on the angle of incidence of the primary electrons on the sample, as long as this angle of incidence is greater than the total-reflection angle.

[0020] In this construction, irradiation with primary electrons having an energy of approximately 1 keV is performed by means of the electron gun 12 while a sample is deposited on the surface of the substrate 11 by vapor deposition using the vapor deposition apparatus 13. In this case, the absorption current that is measured in the substrate 11 oscillates with the period of a monatomic layer as a function of the film thickness of the sample that is formed on the

surface of the substrate 11. The conditions of this oscillation are the same as the conditions of the oscillation of the secondary electron intensity shown in Figure 2. Like the period of the oscillation of the secondary electron intensity, the period T of this oscillation is equal to the thickness of a monatomic layer of the sample; accordingly, the thickness of the gold shown on the horizontal axis can be calibrated by T, so that the accurate thickness of the gold thin film can be ascertained. Accordingly, high-precision film thickness measurements can be made by using this phenomenon. In this embodiment, unlike the first embodiment, no secondary electron detector is required; accordingly, a simpler film thickness measurement apparatus can be realized.

[0021] Furthermore, as in the case of the oscillation of the secondary electron intensity, there may be instances in which the energy of the primary electrons at which the oscillation of the absorption current appears most conspicuously varies according to the type and surface orientation, etc., of the sample. In the present embodiment, the energy of the primary electrons was set at approximately 1 keV as an example; however, the present method may be used under optimal conditions at which the oscillation of the absorption current appears most conspicuously by varying the energy of the primary electrons in accordance with the type and surface orientation of the sample.

[0022] Figure 5 is a diagram which shows a fourth basic construction of an embodiment of the film thickness measurement apparatus of the present invention. This construction comprises a substrate 11, an electron gun 12 which is used to irradiate this substrate 11 with primary electrons, a vapor deposition apparatus 13 which deposits a sample on the surface of the substrate 11 by vapor deposition, a secondary electron detector 14 which is used to measure the intensity of the secondary electrons that are knocked out [of the sample] by the primary electrons, and a computer 51 which is used to control these devices and to collect data. Furthermore, as in the first embodiment, the electron gun 12 has a function that allows the energy of the primary electrons to be freely set at an energy ranging from approximately 10 eV to approximately 5 keV. Moreover, as in the first embodiment, there are no particular restrictions on the angle of incidence of the primary electrons on the sample, as long as this angle of incidence is greater than the total-reflection angle.

[0023] In this construction, irradiation with primary electrons having an energy of approximately 1 keV is performed by means of the electron gun 12 while a sample is deposited on the surface of the substrate 11 by vapor deposition by means of the vapor deposition apparatus 13. In this case, the intensity of the secondary electrons that are emitted from the surface of the thin film sample that is formed on the surface of the substrate 11 is measured by

the secondary electron detector 13 [*sic*]*. As in the first embodiment, the measured intensity of the secondary electrons oscillates as a function of the film thickness of the sample that is formed on the surface of the substrate 11, and since the period T of this oscillation is equal to the thickness of a monatomic layer of the sample, high-precision film thickness measurements can be performed using this oscillation. Film thickness measurement can be automated by monitoring the signal of this secondary electron intensity oscillation by means of the computer 51.

[0024] Furthermore, in this embodiment, as in the first embodiment, there may be cases in which the energy of the primary electrons at which the oscillation of the secondary electron intensity appears most conspicuously varies according to the type and surface orientation, etc., of the sample. Accordingly, in this embodiment as well, the present method may be used under optimal conditions at which the oscillation of the intensity of the secondary electrons appears most conspicuously by adjusting the energy of the primary electrons in accordance with the type and surface orientation of the sample. In this case, automation can be accomplished by performing the measurement of the oscillation of the intensity of the secondary electrons and the control of the electron gun 12 by means of the computer 51.

[0025] Furthermore, in the present embodiment, control of the vapor deposition rate can be automated by monitoring the film thickness with respect to the vapor deposition time by means of the computer 51, and controlling the vapor deposition apparatus 13 so that the vapor deposition rate is maintained at an arbitrary rate.

[0026] Furthermore, in the present embodiment, the manufacture of samples with an arbitrary film thickness can be automatically performed by monitoring the film thickness with respect to the vapor deposition time by means of the computer 51, and automatically stopping vapor deposition using the computer 51 when the desired film thickness is obtained. In this case, information regarding the desired film thickness may be input into the computer 51 beforehand.

[0027] Furthermore, there may be cases in which the secondary electron energy range in which the oscillation of the intensity of the secondary electrons appears most conspicuously varies according to the type and surface orientation, etc., of the sample. Accordingly, in this embodiment as well, as in the second embodiment, it is possible to utilize the energy analyzer so that only secondary electrons that have a specified energy are conducted to the secondary electron detector 14, and so that the intensity of these secondary electrons is measured. This energy discrimination can also be automated by means of the computer 51.

* Translator's note: apparent error in the original for "secondary electron detector 14."

[0028] Furthermore, the oscillation of the intensity of the secondary electrons was utilized in the present embodiment; however, it would also be possible to utilize the oscillation of the absorption current indicated in the fourth embodiment. In this case, the secondary electron detector 14 becomes unnecessary, so that the construction of the apparatus as a whole is further simplified.

[0029] Furthermore, in the above description, the irradiation of the sample with an electron beam was performed over the entire surface of the sample; however, [the present invention] can be similarly worked in cases where the electron beam is constricted, so that the film thickness in a specified region of the sample is measured.

[0030]

[Effect of the Invention] In the present invention, the film thickness of a sample during film formation by vacuum deposition can be measured and controlled with a precision corresponding to the thickness of a monatomic layer or better by irradiating the sample with the angle of incidence of the primary electrons set at an angle that is greater than the total-reflection angle. Accordingly, the following effects are obtained: namely, the construction of the overall apparatus can be simplified, handling of the apparatus is facilitated, and the cost of the overall apparatus can be reduced. In the present invention, furthermore, a low-energy electron gun whose structure is relatively simple compared to that of a conventional device using reflective high-speed electron diffraction is used. Accordingly, the following merit is also obtained: namely, the overall apparatus comprises an inexpensive system compared to cases where a special high-voltage power supply, electron gun and electron-optical system are used. The applicability of these effects in scholarly fields, and the industrial value of these effects, are extremely high.

[Brief Description of the Drawings]

[Figure 1] Figure 1 is a diagram which shows the basic construction of the present invention.

[Figure 2] Figure 2 is a diagram of the oscillation of the secondary electron intensity that is obtained in the present invention.

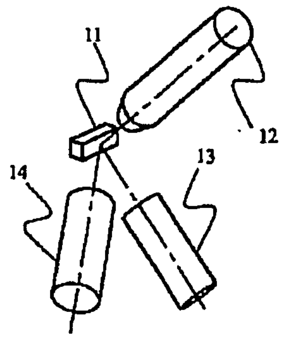
[Figure 3] Figure 3 is a diagram showing the combination of an energy analyzer with the present invention.

[Figure 4] Figure 4 is a diagram showing the construction used in a case where the oscillation of the absorption current is used in the present invention.

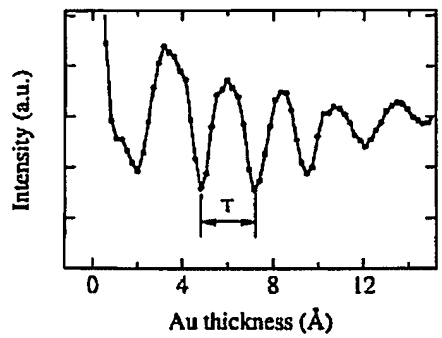
[Figure 5] Figure 5 is a diagram showing the automatic control of vapor deposition by the present invention and a computer.

[Explanation of Symbols]

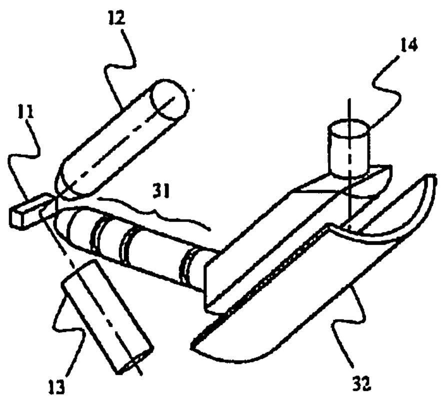
11... Substrate; 12... Electron gun; 13... Vapor deposition apparatus; 14... Secondary electron detector; 31... Electron lens; 32... Energy analyzer; 51... Computer.



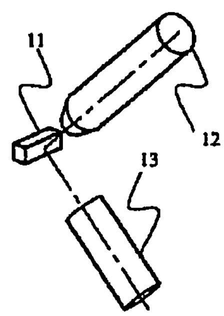
[Figure 1]



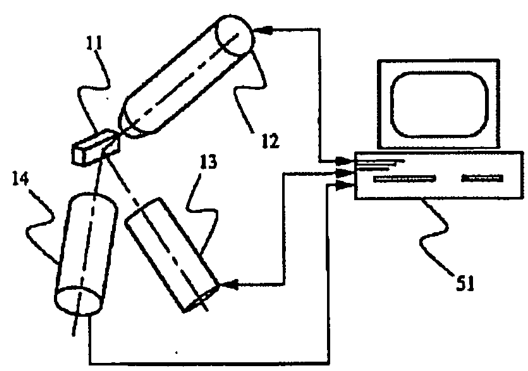
[Figure 2]



[Figure 3]



[Figure 4]



[Figure 5]